KERNFORSCHUNGSANLAGE JÜLICH GmbH

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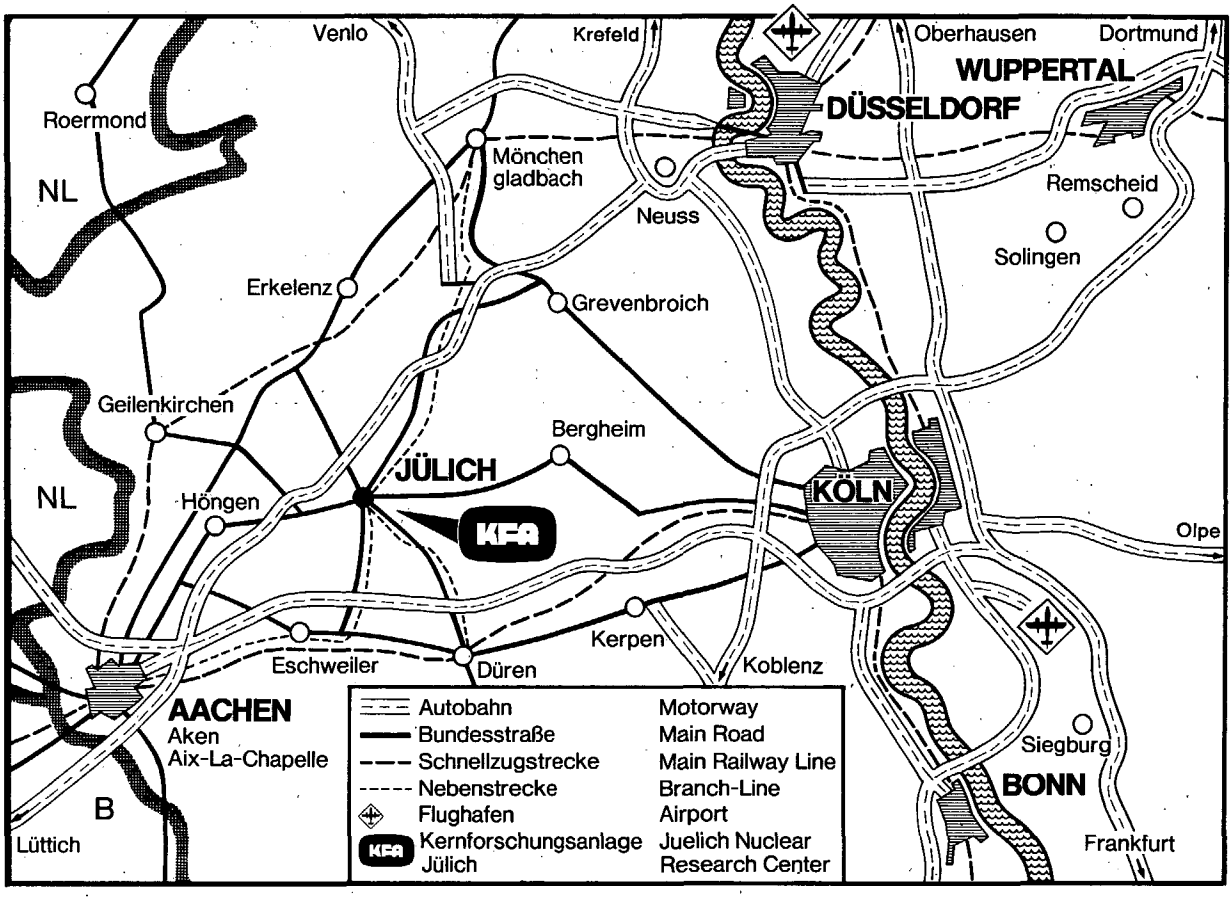
W. W. Delle, K. Koizlik, H. Luhleich, H. Nickel

Jül - 1333
August 1976

Als Manuskript gedruckt



Berichte der Kernforschungsanlage Jülich – Nr. 1333

Institut für Reaktorwerkstoffe Jül - 1333

Im Tausch zu beziehen durch: ZENTRALBIBLIOTHEK der Kernforschungsanlage Jülich GmbH, Jülich, Bundesrepublik Deutschland

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ABSTRACT

The growing use of nuclear reactors for the production of electric power throughout the world, and the consequent increase in the number of nuclear fuel manufacturers, is giving enhanced importance to the consideration of quality assurance in the production of nuclear fuels. The fuel is the place, where the radioactive fission products are produced in the reactor and, therefore, the integrity of the fuel is of utmost importance. The first and most fundamental means of insuring that integrity is through the exercise of properly designed quality assurance programmes during the manufacture of the fuel and other fuel element components.
The International Atomic Energy Agency therefore conducted an International Seminar on Nuclear Fuel Quality Assurance in Oslo, Norway from 24 till 28 May, 1976. The seminar was addressed to a wider audience and was, with its educational content, of interest in those countries newly entering the field as well as in those with established nuclear fuel fabrication capabilities.
This KFA report contains a paper which was distributed preliminary during the seminar and - in the second part - the text of the oral presentation. The tables and figures were presented as slides.
The paper gives a summary of the procedures available in the present state for the production control of HTGR core materials and of the meaning of the particular properties for reactor operation.

The work was partly sponsored by the Bundesministerium für Forschung und Technologie of the Federal Republic of Germany as well as by the Government of North-Rhine-Westfalia.

Kernforschungs-
anlage Jülich
GmbH IRW

JÜL - 1333

August 1976

VERFAHREN ZUR QUALITÄTSKONTROLLE FÜR HTR BRENNELEMENT-KOMPONENTEN

von

W.W. Delle
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H. Nickel

KURZFASSUNG

Die wachsende Verwendung von Kernreaktoren für die Produktion von elektrischer Energie in der Welt und die wachsende Anzahl der Brennelementhersteller bedingen eine zunehmende Bedeutung der Qualitätssicherung bei der Produktion von Brennelementen. Der Brennstoff ist die Stelle, an der die radioaktiven Spaltprodukte im Reaktor entstehen. Deshalb ist es von fundamentaler Bedeutung sicherzustellen, daß die Produktion von Brennstoff und Brennelementen von einem genau ausgearbeiteten Qualitätsprogramm begleitet ist. Die Internationale Atomenergie-Behörde (IAEA) hat deshalb ein internationales Seminar über die Qualitätssicherung von Nuklearbrennstoff vom 24. bis 28. Mai 1976 in Oslo durchgeführt. Das Seminar wandte sich mit seinem Lehr-Inhalt an einen erweiterten Zuhörerkreis, wobei sowohl Teilnehmer aus Ländern, die neu in das Nukleargebiet einstiegen, als auch solche aus Ländern mit eingeführter Nukleartechnik angesprochen werden sollten.

Der vorliegende KFA-Bericht enthält ein Papier, das als vorläufiges Manuskript an die Teilnehmer verteilt wurde. Der zweite Teil enthält das Manuskript des in Oslo gehaltenen Vortrages. Die Tabellen und Abbildungen wurden als Dias gezeigt. Der Bericht stellt eine Übersicht über die zur Zeit verfügbaren Qualitätsverfahren für HTR-Kernmaterialien dar und behandelt die Bedeutung der einzelenen Eigenschaften für den Reaktorbetrieb.

Die Arbeit entstand zum Teil unter der Förderung des Bundesminsteriums für Forschung und Technologie der Bundesrepublik Deutschland sowie des Landes Nordrhein-Westfalen.

Quality Control Procedures for HTGR Fuel Element Components

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Abstract

High temperature gas-cooled reactors (HTGR) being designed for the steam cycle are also considered to be of interest, because of their development potential, for advanced systems, e.g. use with the direct cycle gas turbine (HHT) as well as the generation of nuclear process heat reactors (NPH) at high temperatures. The use of nuclear process heat for endothermic reactions, such as methane splitting, gasification of coal and olefine production, requires reactor coolant outlet temperatures between 850 and 1000°C. This is substantiated by the results so far achieved in developing a wide range of materials for high temperature reactors. Key factors are the fuel elements and their components, the quality control of which plays an important role for the reactor verification.

The materials used in the HTGR differ in composition, size and shape. Graphite and graphitic matrix materials are produced in blocks, rods or spheres so that conventional procedures can be applied as far as pre-irradiation quality control is concerned. The small specimens used for irradiation experiments require modifications of the standard procedures. For the characterization of the fuel kernels with diameters up to some 100 µm as well as of the pyrolytic carbon and silicon carbide coating layers with thicknesses in the range smaller than 100 µm, new methods had to be developed.

In this paper, the procedures currently applied during quality control of fuel element components are briefly described and their meaning is discussed.

1. Introduction

Among nuclear energy supply systems, high temperature gas cooled reactors (HTGR) offer a remarkable alternative to other reactor concepts. Main advantages are fuel cycle flexibility, significant inherent safety, and a variety of application modes. One of the most important properties of a modern HTGR is its developmental potential towards temperatures near or even above 1000°C making this reactor system applicable to process heat generation. Compared with other reactor concepts, such as LWR, it could be commercially used with the direct cycle helium gas turbine (HHT). Two different fuel element designs for the HTGR are now available: The spherical element and the hexagonal block type element, the latter meanwhile existing in two different varieties: the machined graphite block and the pressed monolith.
These three fuel element designs are based on the same fuel particle concept. Although it has reached a very high standard of perfection, coated fuel particle technology has not yet reached the technical routine of water reactor fuel technology, thus implying the necessity of broad and complex quality control.

2. Fuel element conceptions and components

As already mentioned, three different fuel element concepts are available today, one with spherical fuel elements and two with block-type elements. The spherical as well as the pressed monolithic elements are made from graphitic matrix material.
The third fuel element species is produced by machining graphite blocks.
Two different materials are to be considered, arising from these fuel element concepts: graphitic matrix and reactor graphite. Although being produced from similar raw materials, the final products differ because of the different production histories.

The common link of the fuel element concepts is the coated nuclear fuel particle, comparable to the fuel pellet of a water reactor. It consists of the fuel kernel, some 100 µm in diameter depending on the fuel cycle concept, and the coating,

the main fission product barrier. Thus the coating can be compared with the cladding of water cooled reactor fuel elements. At present,the fuel kernels consist of uranium and thorium oxides or carbides. The kernel is coated by a sequence of pyrocarbon layers. The inner layer adjacent to the kernel is always highly porous, providing the particle with an empty volume for the fuel kernel swelling and the gaseous fission product reception. The outer layers form a pressure vessel and fission product diffusion barrier.
Because of the poor retention of some special fission products such as cesium, strontium and barium,the coating can be supplied with a silicon carbide interlayer as an additional diffusion barrier. Thus the coated particle production is based on three different materials, the quality of which has to be guaranteed: the fuel, pyrocarbon and silicon carbide.

3. Quality Control procedures

The physical characterization procedures applied for reactor material quality control can be divided into three groups:

- procedures for the determination of specific material properties
- quality control during manufacture with feed back to the production
- measuring of irradiation induced changes of the particular properties.

The division for the particular materials is demonstrated in TABLE I.

3.1 Density

Density is one of the most common physical characteristics of a material. Its significance for fuel element components is different for each of the constituents.
The density of f u e l influences both the heavy metal content in a fuel element and the porosity, which is on one hand necessary for the fission gas absorption, but on the other hand reduces thermal conductivity. Furthermore, the swelling under irradiation depends upon the available porosity.

Sec.	Property	Fuel element component to be characterized				Determination of specific material properties			Quality control during manufacture			Determination of irradiation induced property changes		
		fuel kernel	coating layer PyC	coating layer SiC	struct. material	fuel kernel	coating	struct. material	fuel kernel	coating	struct. material	fuel kernel	coating	struct. material
3.1	Density apparent real	x o	x x	x o	x x	x o	x x	x x	x o	x o	x x	x o	x x	o x
3.2	Porosity	x	x	o	x	x	x	x	o	o	o	o	x	x
3.3	Ceramographic sections	x	x	x	x	x	x	x	x	x	x	x	x	x
3.4	Lattice parameters	o	x	o	x	o	x	x	o	o	o	o	x	x
3.5	Orientation anisotropy	o	x	o	x	o	x	x	o	x	x	o	x	x
3.6	Oxidation behaviour	o	x	x	x	o	x	x	o	x	x	o	o	o
3.7	Determ.of U-content	o	x	o	o	o	o	o	o	x	o	o	o	o
3.8	Leach test	o	x	o	o	o	x	o	o	x	o	o	x	o
3.9	Thermal expansion	o	o	o	x	o	o	x	o	o	x	o	o	x
3.10	Heat conduction	o	o	o	x	o	o	x	o	o	x	o	o	x
3.11	Electrical resistivity	o	o	o	x	o	o	x	o	o	x	o	o	o
3.12	Young's modulus	o	x	x	x	o	x	x	o	o	x	o	x	x
3.13	Strengths	o	x	x	x	o	x	x	o	o	x	o	o	x
3.14	Irradiation induced creep	o	o	o	x	o	o	x	o	o	x	o	o	x

x to be characterized
o not to be characterized
structural materials = graphite and graphitic materials

TABLE I: Quality procedures for HTGR fuel element components

The density of p y r o l y t i c c a r b o n is important for fission gas retention and also for irradiation behaviour. There is a tendency to have a high density for good retention. On the other hand, high density reduces the mechanical stability under irradiation. Thus a carefully controlled optimization is necessary.

The main objective of the g r a p h i t e and m a t r i x density is achieving a degree of moderation to obtain a sufficient neutron economy. Density is also a relevant measure for the homogeneity of big graphite blocks. Some other properties such as strength, Young's modulus of elasticity, thermal conductivity and thermal expansion can be related to density.
In general, density is calculated from mass and volume. Mass is straightforward and can easily and accurately be obtained by modern laboratory balances. As far as the ceramic HTGR materials are concerned volume measurements are more difficult to define and attain because they are influenced by variables such as the presence or absence of voids and pores in the solids being measured. An other aggravating condition can consist in the smallness of the specimens to be measured as in the cases of pyrolytic carbon or silicon carbide layer materials. Thus a rather ambiguous vocabulary surrounds the subject of density determination,and it is an important task of quality control institutions to define correctly the various densities.

3.1.1 Apparent density (bulk density, geometrical density)

The apparent density is the mass per unit volume. It is assumed that the volume is homogeneously filled with mass. It includes the volumes of the substance, displacements, micro and macro pores and internal voids.
The apparent density is measured on fuel kernels, coating materials, and graphite. For all these components its measurement is important as a specific material property and quality control procedure during manufacture. The apparent density is of fundamental significance for the irradiation behaviour of fuel and coating materials.

F u e l k e r n e l s : To calculate the mean volume of the spherical fuel particles it is necessary to measure their diameters. A simple technique is based on a count of about 200 kernels lined up in a V-slot. The mean diameter is deduced from the total number. The diameter distribution is determined by microscopic measurements or by X-ray micrographs.

For statistical considerations and on-line quality control an automatic optical particle analyzer developed by ÖSGAE Seibersdorf/Austria is used (1).
A quantity of kernels is moved along a constant light source and a very small measuring gap with intervals of approximately 20 particles per second. The gap area shadowed by a particle is measured by means of a photodiode and is a measure for the diameter. The apparatus is controlled and the data are evaluated by an on-line process computer. This technique is suitable for diameters of 150 to 1500 µm. After weighing the investigated portion of kernels the apparent density can be calculated.

Another method applied is the mercury pycnometer technique. Because of the great surface tension of this liquid the mercury displaced by the fuel material can easily be determined.

The apparent density of p y r o l y t i c c a r b o n and s i l i c o n c a r b i d e can be calculated from the increase in volume and mass just after deposition measured by using the particle analyzer (see fuel kernels).

The apparent density of g r a p h i t e and g r a p h i t i c m a t r i x materials can be determined by measurements of mass and linear dimensions.
The determination can also be done using the displacment of water, xylene or mercury. Using water or xylene, the specimens have to be saturated with these liquids. Then they are measured in the liquid and in air by use of a hydrostatic balance. The difference of both weighings (buoyancy) corresponds to the volume of the specimen (2).

3.1.2 Sink-float density

The purpose of this method is to determine the density of p y r o l y t i c c a r b o n layers by suspension in a density gradient column. The gradient column consists of isobutanol and bromoform chemicals mixed continuously in varying proportions so as to cover a range of densities from 0.8 g . cm^{-3} to 2.8 g . cm^{-3}. Pyrolytic carbon fragments cracked from coated particles are suspended into the column. The density is defined at the point in the column where the test specimen of pyrolytic carbon has reached the same density as the liquid in which it is suspended (fig. 2).
Using other liquids the sink-float method can also be employed for the determination of the density of s i l i c o n c a r b i d e layers. If the layers are impermeable for the liquids applied the values of apparent and sink-float density are equal.
This method can be modified by stepwise admixing small defined quantities of one with the other of the liquids mentioned above. In this way the mean density of the mixture is changed step by step, upon reaching the density of the particular pyrolytic carbon the layer floats in the liquid.

3.1.3 Real density (true density, substance density).

Real density is the density as determined when the p y r o - l y t i c c a r b o n or g r a p h i t e has been reduced to a particle size so small that it accomodates no internal voids. This occurs by grinding to particle sizes of about 1 µm. The volume of the powder is determined by using a pycnometer with xylene which is displaced by the matter.
This is a quality control procedure for graphite- and graphitic-matrix-producing firms to check the reproducibility of the raw materials delivered, which is important for the irradiation behaviour.

3.2 Porosity

The porosity of f u e l k e r n e l s is important for the fission product absorption and for the buffering of the swelling under fast neutron exposure and by burn-up. As far as p y r o l y t i c carbon and g r a p h i t e are concerned the transport mechanisms for both solid and gaseous fission products are influenced by porosity and especially by pore size distribution.
Porosity can be correlated with other properties such as thermal conductivity, thermal expansivity, strength and corrosion behaviour.

3.2.1 Total porosity

Total porosity is calculated from apparent and theoretical (or real) density

$$P = 1 - \frac{d_a}{d_{th}}$$

d_a = apparent density
d_{th} = theoretical density

It includes all irregularities such as defects and imperfections. If these effects are neglected, the real density instead of theoretical density can be used for the calculation.

3.2.2 Open porosity

F u e l and g r a p h i t i c materials are evacuated and then impregnated with a suitable liquid (xylene, water) and the increase in weight is measured. Using the specific weight of the liquid the pore volume can be calculated.
Applying gas methods one can measure the outer linear dimensions for volume calculation and the gas displacement by the matter in a graduated vessel. The difference between both volume measurements is the open porosity.
Another possibility is to calculate open porosity from the surface area.

3.2.3 Pore size distribution

In general the mercury penetration method is used. Mercury is pressed stepwise into the open pores and under the assumption of cylindrical pores a mathematical pressure-to-pore diameter relation is used for determining pore sizes.

$$p \cdot r = 2\sigma \cos \Theta$$

p = pressure, r = radius of pores, σ surface tension,
Θ = limiting angle between mercury and specimen

This method is valid for pore diameters of more than 40 Å. Smaller pores can be measured by gas adsorption methods depending upon the gas used. Recently the pore size distribution of macropores in pyrocarbon, graphite and fuel kernels with diameters of more than 0,5 µm as well as total number and total volume of pores have been determined by the quantitative image analyzer, which has a considerable advantage in quality control (3,4).

3.3 Ceramographic sections

Ceramographic sections are relevant for all groups of materials discussed in this paper.
They are necessary to control the size, shape and composition of the kernels, allow an appraisal of the thickness, shape and structure of the pyrolytic carbon and silicon carbide layers and are necessary for the application of quality control procedures such as

- porosity and pore size distribution (measured by
- quantitative image analysis) (3.2.3)
- optical orientation anisotropy (3.5.2)
- plasma and wet-oxidation (3.6.2)

The investigation of grain/binder coke distribution and porosity of graphite again requires ceramographic sections.

The homogeneous distribution of coated fuel particles of fuel rods embedded in the block-shaped graphite fuel elements and in spherical fuel elements is controlled by ceramographic section.
It is a most important characterization method which is applied in the on-line quality control of coated fuel particles and graphitic materials.

3.4 Lattice parameters

Crystallite size and lattice spacing are of importance for the classification of both p y r o c a r b o n and g r a p h i t e because they are an indication of the degree of order in both materials (5). This again influences on the properties as well as the irradiation and chemical behaviour. Both properties should be known for the complete understanding of the materials produced but the determination of these properties is not a procedure to be applied during quality control.
Both crystallite size, L_c, and lattice spacing $c/2$, are measured by means of X-ray diffraction, in the case of $c/2$ being measured up to high temperatures.
The mean dimension of the crystallites composing a powder is related to the true X-ray diffraction broadening, ß, by the equation

$$L = \frac{K \cdot \lambda}{\beta \cos\alpha}$$

where K is a constant approximately equal to unity and related both to the crystallite shape and to the way in which ß, L are defined. λ is the x-ray wave length, ß is the true breadth of the diffraction peak at half the peak height, and α is BRAGG's angle (6, 40)

3.5 Orientation anisotropy

Due to their microstructure the properties of p y r o l y t i c c a r b o n s and g r a p h i t e s are different for the different directions. This orientation anisotropy is highly important for the suitability of these materials in HTGR.

P y r o l y t i c c a r b o n is required to be nearly isotropic, because the heat conduction has to be sufficient in both radial and tangential directions. This can be achieved by an isotropic coating. Under irradiation, the dimensional changes of near isotropic coatings are much smaller than those of anisotropic ones. Furthermore the inner stresses are limited to an acceptable magnitude so that the danger of breaking during reactor operation is reduced considerably. The influence of orientation anisotropy on the dimensional behaviour under irradiation is significant for g r a p h i t e and m a t r i x materials as well. Especially in the case of large blocks, inner stresses are built up which are the greater the larger the dimensions of the block and the higher the orientation anisotropy. In the very small pyrolytic carbon specimens the anisotropy is determined either by X-ray diffraction or by optical bi-reflection.

3.5.1 X-ray diffraction

This method has first been developed and described by BACON (7). By means of the integral intensity J (Ø) a frequency distribution of the interference (002) is determined where Ø is the angle between deposition normal and c-axis of the particular crystallites.

The BACON anisotropy factor then is

$$BAF = 2 \int_0^{\pi/2} J(\Theta) \sin\Theta \cos^2\Theta \, d\Theta \Big/ \int_0^{\pi/2} (\Theta) \sin^3\Theta \, d\Theta$$

The BAF is basicly defined by the coefficients of thermal expansion at 400°C parallel and perpendicular to the preferred orientation.

3.5.2 Optical anisotropy

The BAF cannot be measured easily on coated fuel particles. It is therefore necessary to co-deposit pyrolytic carbon on graphite substrates to get small disks which can be investigated.

But it has been found out that the structure of pyrolytic carbon on spherical particles and plane supports is not homogeneous and equal (8,9). Therefore an optical method was applied basing on the bi-reflection of crystalline graphite. A beam of linear polarized light is directed on a ceramographic section through the equatorial area of the coated particle. The intensity of the reflected light is measured when both the polarization and the preferred direction are parallel to one another and when they are in the perpendicular situation. The ratio of both is the optical anisotropy factor OPTAF or OAF (10,11,12) (fig. 2).

3.5.3 Anisotropy of thermal expansivity

The anisotropy of g r a p h i t e specimens is commonly determined by the ratio of thermal expansivity parallel and perpendicular to grain orientation. To save time the electrical resistivities in both directions can be used as well. But the results obtained from both properties do not always agree well so that the information about each measured property is necessary (see TABLE II).

Material / Anisotropy	Moulded gilso-carbon graphite	Extruded petroleum coke graphite	Extruded natural graphite
Thermal expansivity	1,07	1,27	5,06
Electrical resistivity	1,06	1,24	3,44

TABLE II: Examples for anisotropies of graphites

The anisotropy of graphitic materials is highly important for the irradiation behaviour so that its production control with statistical evaluation as well as knowledge of its irradiation-induced change are necessary (41).

3.6 Oxidation behaviour

The coolant gas in HTGR, helium, is not as clean as was assumed earlier. Water vapor is present and forms a potential for oxidizing both g r a p h i t e and c o a t i n g layers which can lead to failure because of the decrease in density and strength. The oxidation can be limited by the raw materials used and the thermal history during production (31).
Quality control is therefore indispensable.
Because graphite and pyrocarbon are not uniform in structure, but consist of well and poorly ordered areas, it is very important to investigate the composition of these materials. Radiation damage mainly occurs in the well ordered crystalline phase which cannot be oxidized as well as the poorly ordered phase being graphitized during irradiation.

3.6.1 Oxidation by water vapor

Oxidation experiments must be carried out as a routine procedure, the conditions being dependent upon the particular type of HTGR. Currently the procedure is to expose g r a p h i t e s and g r a p h i t i c m a t r i c e s to 1 vol% H_2O in He of 1 bar at 900 or 1000°C for 10 hours. The changes in mass and strength as well as in properties like specific surface area are measured (31).

3.6.2 Plasma- and wet oxidation

The different oxidation behavior of the two components in g r a p h i t e and p y r o l y t i c carbon are used for the characterization especially of near isotropic pyrocarbon layers. Ceramographic sections of pyrocarbon coatings are etched by means of an oxygen plasma or an oxidative solution (13,14,15). Both of these procedures are necessary although the results are similar. During the wet oxidation process only a zone near the surface is etched whilst by the plasma-oxidation method it is possible to detect deeper areas as well. By these methods the distribution of the poorly ordered component

called CBC (carbon black-like component) and its magnitude can be obtained. For evaluation under a microscope it is also possible to use the quantitative image analyzer (see 3.2.3).

3.7 Uranium contamination

During deposition in fluidized beds, traces of uranium are embedded into the p y r o c a r b o n coating layers. Since this uranium content leads to an additional fission product release, the amount of uranium contamination is specified and thus has to be controlled. Two methods are applied for the uranium determination, the α-count and the mechanical grinding method (16).

3.7.1 α-count method

This technique is able to detect the uranium contamination near the surface. For this purpose one determines the α-radiation of a single layer of 100 particles between two Si-semiconducting crystals inside a closed chamber under vacuum. The α-range in high dense pyrocarbon is about 15 µm.

3.7.2 Grinding method

Concentration profiles in the outer layers of coated particles are determined by means of the grinding method.
The coatings of coated particles are ground stepwise in special glass containers. These containers are cylindrical glass tubes with a hemispherical bottom and inner walls having ROUGH surfaces. Four of these glass containers are fixed into a mill and the particles are ground by rotation.

The uranium concentration of the abraded pyrocarbon is determined by a microanalytical method. At first the pyrocarbon is completely oxidized by a mixture of nitric and sulfuric acid under reflux. After evaporation of the acids the residue is dissolved in water and a pill of sodium and lithium fluoride is impregnated with this solution.

The uranium content is then determined by a fluorimetric method (16).

3.8 Leach test

The leach test is a rough method for the detection of microcracks in pyrocarbon coating layers (17). A sufficient number of coated particles is leached in a glass bulb at a temperature of 95°C for 8 h with Thorex reagent, consisting of 8 n - nitric acid mixed with concentrated fluoric acid. For the fluorimetric determination of the uranium content the solution is handled in the same way as described in chapter 3.7.2 .
If the kernel consists of uranium and thorium, the thorium is coverted into Pa 233 by neutron irradiation and determined by γ-spectrometry (activation analysis).

3.9 Thermal expansion

Thermal expansion of fuel particles as well as pyrocarbon and silicon carbide layers can be neglected because of the small size of these components. Furthermore, under irradiation dimensional changes occur, which are much more significant than thermal expansion. For g r a p h i t e fuel elements and reflector blocks, it is important to know the coefficient of linear thermal expansion, C.T.E., since there is a broad range of values from $1.5^{10-6}/°C$ to about $6 \cdot 10^{-6}/°C$ depending on the raw materials used, pressing methods applied and heat treatment history (18). Under irradiation, the C.T.E. changes.Depending upon fluence and irradiation temperature as well as crystallite size and orientation anisotropy the C.T.E. can increase or decrease (18,19).
From these observations it follows that the C.T.E. is a property which must be determined, because of its relevance for the orientation anisotropy. Furthermore, its irradiation behaviour has to be determined, because of its importance for the design and for calculations which give information

on the stresses under operation and reactor shut-down conditions.

The C.T.E. is measured in dilatometers, commonly in the range between room temperature and 500 or 1000°C. The expansion is transferred to an optical system or to an inductive linear transducer by means of silicon push rods. From the slope of the temperature-dilatation curve the C.T.E. ist calculated.

3.10 Heat conduction

Knowledge of heat conduction of reactor core materials is of high priority. However, the small size of the fuel kernels (150-1000 µm) and the thin layers of pyrolytic carbon or silicon carbide (50-100 µm) make it unnecessary to measure this property, although, it has been shown that the thermal conductivity of packed beds of coated fuel particles depends upon the density and thickness of the layer as well as upon the size and size distribution of the coated fuel particles (assuming the anisotropy of the coatings compared is about 1) . Of course a further improvement is achieved by bondings or compacts.
Heat conduction of graphites and graphitic matrix materials should be measured after the development of a new product to complete the data set. It is important to measure heat conduction up to about 1000°C. It is not necessary to be applied as a routine control. The determination of the irradiation induced changes should not be neglected.

3.10.1 Thermal conductivity

Axial and radial heat conduction are used to determine thermal conductivity of graphites. The procedure to be chosen depends upon the particular case. For example, the investigation of specimens cut from large blocks allows one to work with large samples, whilst for irradiation experiments it is necessary to measure small samples (20).
The advantage of measuring the heat conduction in the axial direction is the possibility of using standards for comparison, but there is the disadvantage of heat losses by radiation, especially at high temperatures. Ref. (20) describes how

accurate measurements can be carried out in spite of heat radiation. Using radial heat transport, heat losses are avoided but there is the problem of achieving strict radial heat flux because of the existence of axial temperature gradients.

3.10.2 Thermal diffusivity

Thermal diffusivity is measured on small disks, which is one of its advantages . Another is the fact that the measurement can be carried out in a very short time since it is not necessary to adjust to steady state conditions as in the case of thermal conductivity.
Two variations of this test method are in use

- a pulse method applying a periodic heat pulse at one end of the specimen (21,22)
- a flash method; a light flash is directed on the specimen, and the increase in temperature at the back side of the sample is measured (23,24).

3.11 Electrical resistivity

Electrical resistivity is of no relevance for nuclear reactor materials with respect to reactor operation. But in the case of g r a p h i t i c materials it is applied to control the degree of graphitization. Sometimes this property is measured instead of thermal conductivity for quality control purposes . Although at room temperature the electronic portion of heat conduction is less than ten percent, this procedure is valid for a group of materials produced from the same constituents.
Electrical resistivity can be measured either by the 4 point method (voltage, current) or by means of the Wheatstone's bridge. It is a fast method for quality control and gives a reliable information on irradiation damage in graphite.

3.12 Youngs's modulus of elasticity

Young's modulus is a property of interest for p y r o c a r b o n and g r a p h i t e . For these materials it can be related to strength, which has the advantage that in the case of

g r a p h i t e the measurements can be carried out using non-destructive methods (31). Because Young's modulus is very sensitive to local variations in large graphite blocks, it is suitable for distribution characteristics in production charges. It has been found that the pre-irradiation Young's modulus is significant for the irradiation induced dimensional changes (25,26,27). Owing to this ,this property is of excellent meaning for grahitic materials which are to be used in HTGRs.

3.12.1 Dynamic method

A longitudinal resonance frequency f (fundamental oscillation) is generated in the g r a p h i t e specimen. This frequency is related to other important quantities by the relation

$$E = 4 \cdot L^2 \cdot f^2 \cdot d$$

where E = Young's modulus
L = length of specimen
d = apparent density

3.12.2 Static method

In the case of the three-point method, the g r a p h i t e specimen is used as a beam supported on both ends. The load is applied in the middle between both supports. Using the four-point method the load acts on two points having a load span of at least two times the sample thickness and the support span three times the load span (2, 28).

3.12.3 Ring test method (whole and half ring)

Specimens of p y r o c a r b o n and s i l i c o n c a r b i d e prepared from coated fuel particles cannot be measured by means of the procedures described under 3.12.2 because of their small size. Therefore the ring test method is applied. The rings or half rings are pressed on both sides of their diameter in the ring pressing apparatus consisting of

two parallel sapphire plates. The deflection of the ring and the force applied are measured (2, 29) (fig. 2)

3.12.4 Coated particle test method

If the coated fuel particle has no silicon carbide layer so that there is no support of the kernel the entire particle can be pressed to get the load-deflection curve which is the basis of this method. This procedure can be used for production quality control (30).

3.13 Strengths

In an HTGR the support, reflector and fuel element graphite blocks have to have a sufficient strength. This property is changed under irradiation (increase at lower fluence, decrease at very high fluence) (27,31).
Therefore it is necessary to specify a minimum magnitude of the strength for a particular component and to determine the post-irradiation strengths. Sufficient strength of pyrocarbon and silicon carbide coatings is necessary to withstand the fuel element production with its pressing and heating steps and the fission gas pressure during irradiation. Irradiation induced second order stresses require additional strength. Thus it is obvious that strength measurements have to be carried out to characterize the materials after production as well as before and after irradiation up to life time fluence at temperatures relevant for the particular reactor component.
It seems unnecessary to carry out strength measurements up to irradiation temperatures because the increase at 1000°C is not more than about 20% compared with values at room temperature of measurements on non-irradiated materials (19).

3.13.1 Flexural strength (bending strength)

The equipment is described under 3.12.2. The load is moved down until the specimen breaks (ultimate flexural strength). During deformation no shock is applied on the specimen.
If the fracture occurs within the span length between the

surfaces the flexural strength can be calculated as follows:

$$S = \frac{P\,L}{bd^2}$$

where P = maximum applied load
L = support span length
b = average width of specimen
d = average thickness of specimen

3.13.2 Tensile strength

When using a tensile testing machine, extreme care should taken that the axis of the test specimen is located in the centre line of the testing machine (32). Tensile strength is calculated by dividing the maximum load carried by the specimen during a tension test by the cross-sectional area of the specimen.

3.13.3 Rupture strength

Rupture strength is measured on p y r o l y t i c carbon and s i l i c o n c a r b i d e rings and half rings. The equipment is described under 3.12.3. The rings or half rings are pressed on to the sides of their diameter in the ring-pressing apparatus until they break(Fig. 2). Load-deflection curve and breaking load are plotted. The complicated calculations of ring or half ring rupture stress as well as of the equivalent strength of the spherical shell in the case of coated fuel particles are described in (29).
For unsupported c o a t e d f u e l p a r t i c l e s without a silicon carbide layer the estimate of rupture stress in as-produced particles is possible (33). This means an advantage for quality control of production batches.

3.14 Irradiation induced creep coefficient

Without irradiation, g r a p h i t e normally shows very limited plasticity at temperatures encountered in the core

of the HTGR. Small irreversible deformation of graphite and pyrolytic carbon under stress leads to the well-known permanent set, but the deformation ceases already very early. Remarkable creep strains up to some 60% in graphite at very high temperatures (above 2000°C) have been reported (34). At the very beginning of irradiation in an HTGR, thermal stresses in graphite blocks will occur especially at locations with large temperature gradients. The stress pattern is altered later by the differential WIGNER strain due to both temperature and neutron flux gradients. The graphite block design, therefore, requires some relief of the internal stresses by irradiation induced plasticity or creep. These internal stresses might be detrimental to the integrity of graphite blocks if creep were not taking place. For realistic stress calculations, a reasonably good knowledge is necessary of the irradiation creep in reactor graphites and graphitic matrix materials as a function of temperature, fast neutron flux and fluence up to lifetime of each particular core component. There are several arrangements for determining creep coefficients of graphite.

Constant stress creep experiments are performed using in-pile or out-of-pile strain registration (27,35,36,38).
For creep experiments under varying stress the tube bowing and the restrained shrinkage creep experiments are applied (39), the latter can be carried out without too much expense and permits the investigation of a number of specimens in one irradiation experiment.
In the temperature range between 350 and 1400°C in which all graphites initially shrink, dumb-bell shaped specimens surrounded by split sleeves made of special graphite with a lower shrinkage rate are used (39).
Stresses, tensile in the specimen and compressive in the restrainer, are built up during irradiation because the two components are coupled mechanically, so that they are unstrained to have the same dimensions.

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Part 2: Text of the presentation

During previous 20 years a large number of power reactors has been installed in many countries, most of them being water reactors. But there was also a permanent increase in nuclear power reactors basing on the gas-graphite system, especially in Great Britain and France.
The power of water reactors and AGR could be increased from about one or two hundred MW to far more than 1.000 MW per unit. Proven-type reactors are now technologically mature and have attained industrial-type size.
For fuel element fabriaction, an efficient industry is available so that no significant industrial problems are expected to raise. The high degree of reliability required in all the stages that make up fuel element fabrication involves besides significant process parameters and conditions also a high degree of quality control basing on well defined specifications
In recent years, thanks to increased production and more sophisticated automatic control techniques a high perfection in manufacture and quality assurance could be obtained. The recent introduction of quality assurance standards made it possible to fit the control of the properties of LWR fuel element components.
A similar situation prevails in the gas-graphite and advanced gas reactor development.
The question arises, why the situation of the as proven-type designated reactor systems are discussed at the beginning of this paper dealing with HTGR quality control.
The answer is that there are two significant differences in the high temperature gas-cooled reactor field compared to those of the well established reactor types:

- fuel elements are completely different from those of other reactor types, which means completely new quality control procedures,

- up to now no well established HTGR-system exists, but two prototype power stations of 300 MWel each will probably be under operation between 1976 and 1980.

The first statement concerning HTGR fuel elements shall be explained by means of fig. 1 which is a schematic presentation of the thermal reactor fuel element components.

The core contains the fissile material in a suitable form and also a moderator to slow down the neutrons. The relative amounts and nature of the fuel and moderator determine the energy of the neutrons causing most of the fissions. The solid and gaseous fission prodcuts are retained by the cladding material to prevent radiotoxicity in the cooling circuit.
The core is surrounded by the reflector of a material which is largely dependent on the neutron energy mentioned above. The combination of core and reflector, together with other components present, e.g. coolant and structure, must be capable of maintaining a fission chain.
The various materials used for the particular core components are compared in TABLE III.
Because of the very high temperatures metals cannot be used in the core. This is the fundamental reason for the completely new concept for the HTGR fuel elements. Ceramic materials had to be used for all the components including cladding. Therefore new techniques had to be developed to clad the fuel. Following the concepts of prismatic fuel elements from LWR and AGR it had initially been tried to use graphite densified by pyrolytic carbon deposition in the pores.
But this material failed under irradiation because of the high anisotropy of that pyrolytic carbon. Therefore the development of fuel in the form of microspheres and combined with that the desposition of pyrolytic carbon on these kernels was started.
While the LWR and the AGR could up to a certain extent, use the consisting quality control procedures and standards it became necessary to develop new procedures for the characterization of the very small coated fuel particles used in HTGR.

Because of the small size of these particles unusual procedures had to be applied to get property values which may be equal to those obtained from classical methods on bulk materials.
This shall be demonstrated by the examples density, anisotropy and strength determination of pyrolytic carbon (fig. 2).
The density of p y r o l y t i c c a r b o n is important for the fission gas retention and also for the irradiation behaviour. There is a tendency to have a high density for good retention. But on the other hand, high density reduces the mechanical stability under irradiation. Thus an optimization to be controlled carefully is necessary.
How can the density of pyrolytic carbon layers with a thickness of less than 100 µm be measured with high accuracy ?
The apparent density of p y r o l y t i c c a r b o n can be calculated from the increase in volume and mass just after deposition measured by using the particle size analyzer.
A quantity of kernels is moved along a constant light source and a very small measuring gap with intervals of approximately 20 particles per second. The gap area shadowed by a particle is measured by means of a photodiode and is a measure for the diameter. The apparatus is controlled and the data are evaluated by an on-line process computer. This technique is meanwhile suitable for diameters of 150 to 1500 µm . After weighing the investigated portion of kernels the apparent density can be calculated from mass and volume.
However, after deposition of the following layer the original density has been increased because of carbon penetration into the pores. Therefore the sink-float method, which is in use since about 15 years, is applied after fabrication.
The purpose of the sink-float method is to determine the density of p y r o l y t i c c a r b o n layers by suspension in a density gradient column. The gradient column consists of isobutanol and bromoform chemicals mixed continuously in varying proportions so as to cover a range of densities from 0,8 g . cm^{-3} to 2,8 g . cm^{-3}. Pyrolytic carbon fragments cracked from coated particles are suspended into the column. The density is defined at the point in the column where the test specimen of

pyrolytic carbon has reached the point of the same density of the liquid, in which it is suspended. Using other liquids the sink-float method can also be employed for the determination of s i l i c o n c a r b i d e layers (fig. 2).
This method can be modified by stepwise admixing small defined quantities of one with the other of the liquids mentioned above. In this way the mean density of the mixture is changed step by step; upon reaching the density of the particular pyrolytic carbon the layer floats in the liquid.
If the layers are impermeable for the liquids applied the values of apparent and sink-float density are equal.
But in fact they have open pores so that the results obtained from this method do not agree with those from other procedures. Therefore this specific procedure is called sink-float density though there is no strong definition for it. The main reason is that the sink-float density depends upon the portions of closed and open pores which vary due to the scattering in deposition conditions such as kind of deposition gas, gas concentration, deposition temperature and others.
For high density pyrolytic carbon the sink-float and apparent density values are nearly equal.
Due to their microstructure the properties of p y r o l y t i c c a r b o n s and g r a p h i t e s are different for different directions. This anisotropy is highly important for the suitability of these materials in HTGR.
P y r o l y t i c c a r b o n is required to be nearly isotropic, because the heat conduction through the coating has to be sufficient in both radial and tangential directions. This can be achieved by an isotropic coating. Furthermore, the inner stresses introduced by irradiation are limited to an acceptable magnitude so that the danger of breaking during reactor operation is reduced considerably. The influence of orientation anisotropy on the dimensional behaviour under irradiation is significant for g r a p h i t e and m a t r i x materials as well. Especially in the case of large blocks, inner stresses are built up, which are the greater the larger

the dimensions of the block and the higher the orientation anisotropy.
The influence of anisotropy of graphitic matrix materials for moulded blocks and semihydrostatically pressed spheres on the dimensional changes is demonstrated in fig. 3. It shows that by increasing anisotropy of a material at constant raw materials composition the dimensional changes become more anisotropic. This means that the inner stresses mentioned above increase as well.
Fig. 4 shows the influence of orientation anisotropy on the dimensional changes of unstressed pyrolytic carbon deposited from methane on graphite disks (not on fuel kernels). The greater the pre-irradiation anisotropy was the greater and the more anisotropic the dimensional changes. Because of the shell shape of coated fuel particles these changes do not occur, but the prevention results in a rise of stresses being anisotropy dependent. This means that there is the danger of breaking and combined with that a release of fission products if the anisotropy is higher than about 1,1.
Therefore, the anisotropy of both graphite and pyrolytic carbon is an important objective for quality control.
The orientation anisotropy of g r a p h i t e specimens is commonly determined by the ratio of thermal expansivity parallel and perpendicular to grain orientation. To save time the electrical resistivities in both direction can be used as well. But the results obtained from both properties do not always agree well so that the information about the measured property is necessary. Because of the very small size and the spherical shape of the pyrolytic carbon shells none of these procedures is applicable. Therefore orientation anisotropy of pyrolytic carbon is determined either by X-ray diffraction or by optical bi-reflection (fig. 2). Since it is highly difficult to measure anisotropy on coated particles by means of X-ray diffraction for quality control purposes the application of an optical method became necessary.
A beam of linearly polarized light is directed on a ceramographic section through the equatorial area of the coated particle.

The intensity of the reflected light is measured when both the polarization and the preferred direction are parallel to one another and when they are in the perpendicular situation. The ratio of both intensities is the optical anisotropy factor OPTAF or OAF. Special problems arise from the necessity of determination of mechanical properties not only of graphite, but also of the coatings deposited on the fuel kernels. The Young's modulus and the strength of p y r o c a r b o n and s i l i c o n c a r b i d e prepared from coated fuel particles cannot be measured by means of classical methods because of their small size. Therefore the ring test method is applied. Whole or halfrings are pressed on to both sides of the diameter in the ring pressing apparatus consisting of two parallel sapphire plates. The deflection of the ring and the force applied are measured. The load-deflection curve and breaking load are plotted (fig. 2).
If the coated fuel particle has no silicon carbide layer so that there is no support by the kernel the entire particle can be pressed to get the load-deflection curve which is the basis for this method. This procedure can be applied to production quality control.

At the beginning of my presentation I made the second statement saying that no well established high temperature gas-cooled reactor system exists.
In fact upt to now the fuel elements for three experimental and two prototype HTGR have been or are produced.
Two different approaches so far have been made with regard to the core structure and arrangement of fuel elements in high temperature reactors. The reactor concept developed in the FRG uses spherical fuel elements (6 cm in diameter) arranged in pebble beds to form the core. The AVR test reactor is being operated with about 100.000 fuel elements; the thorium high temperature prototype reactor (THTR), however, requires approcimately 700.000 of these spherical elements. Rod-type fuel elements were used in the two experimental reactors Dragon and Peach Bottom. For further concepts, e.g. the Fort St. Vrain prototype reactor, hexagonal block-type fuel elements have been adopted.

The spherical elements have been developed and are fabricated by the NUKEM/HOBEG Company by a moulding process and represent composite systems of a homogeneous particle-matrix core with a fuel free outer zone.
The GAC element consists of a hexagonal graphite block (height 793 mm, width across flats 360 mm) with normally one coolant channel being hexagonally surrounded by 6 fuel holes. The fuel rods consist of a particle-matrix mixture. All fuel elements are quality controlled by these firms. Together with the research centres the procedures are further developed and improved.
An advantage of the HTGR is that, different from other reactor types, HTGR's are not only suited for steam cycle systems, but also for providing heat at temperature levels beyond 800°C. This permits a direct coupling of a HTGR plant with a helium turbine and also the operation of HTGR plants as nuclear heat units for carying out endothermic chemical processes.
In the AVR, a cooling gas outlet temperature of 950°C has been maintained since February 1974, without a significant increase in the cooling gas activity, which has been surprisingly low for several years; of course, this AVR test is not yet fully representative for a continuous operation with large power reactors, but in view of the results obtained so far with the AVR, there is a justified hope that high cooling gas temperatures are also feasible in large HTGR plants with pebble bed core, without a significant modification of the fuel element.
Moreover, studies by HRB and GAC have generally indicated that, besides the spherical fuel elements, the block-type elements are also capable of achieving helium temperatures of 950°C by increasing the number of cooling holes and fuel zones to improve heat transfer.
In the Federal Republic of Germany, the development of HT power reactors for nuclear process heat and steam generation is under way. Combined with this development is the improvement and development of quality control procedures. Round robin experiments are carried out to compare the results obtained from the quality

control methods which I tried to describe in this paper. First steps have been made to develop and introduce new standards which may help the licencing authorities for their decisions.

Summary:

- For the characterization and quality control of HTGR fuel element components new characterization methods have been developed, especially for the very small coated fuel particles,

- the question of over- or under-determination of the properties investigated is still under discussion and should be the aim of proper irradiation experiments to close the loop reliability-fabrication,

- the standardization of quality control procedures for HTGR is an important problem which has been developed in close collaboration with the DRAGON Reactor Project and now the work still to be done has been clearly identified, and we in the Federal Republic of Germany are proceeding along this line,

- the relevance and the accuracy of quality control during and after production is a premise for the successful commerlization of high power HTGR for various applications.

Reactor System	Maximum Core Outlet Temperature (°C)	Fuel			Cladding		Fuel Element Shape	Moderator	Coolant	Reflector
		Material	Shape	Size (mm)	Material	Thickness (mm)				
LWR	~ 300	UO_2	pellet	~9Φ x 11	Zry	0,7 - 0,9	prismatic	light water		
AGR	680	UO_2	pellet	14,5Φ x 14,5	stainless steel	~ 0,4	prismatic	graphite	CO_2	graphite
HTGR	950	(U Th) O_2	micro-sphere	0,4 - 1Φ	PyC	$\leq$ 0,1	block	graphite	He	graphite
		UO_2 + Th O_2			SiC	$\leq$ 0,08	sphere			
		UC_2								

TABLE III:
Components of various nuclear reactor systems

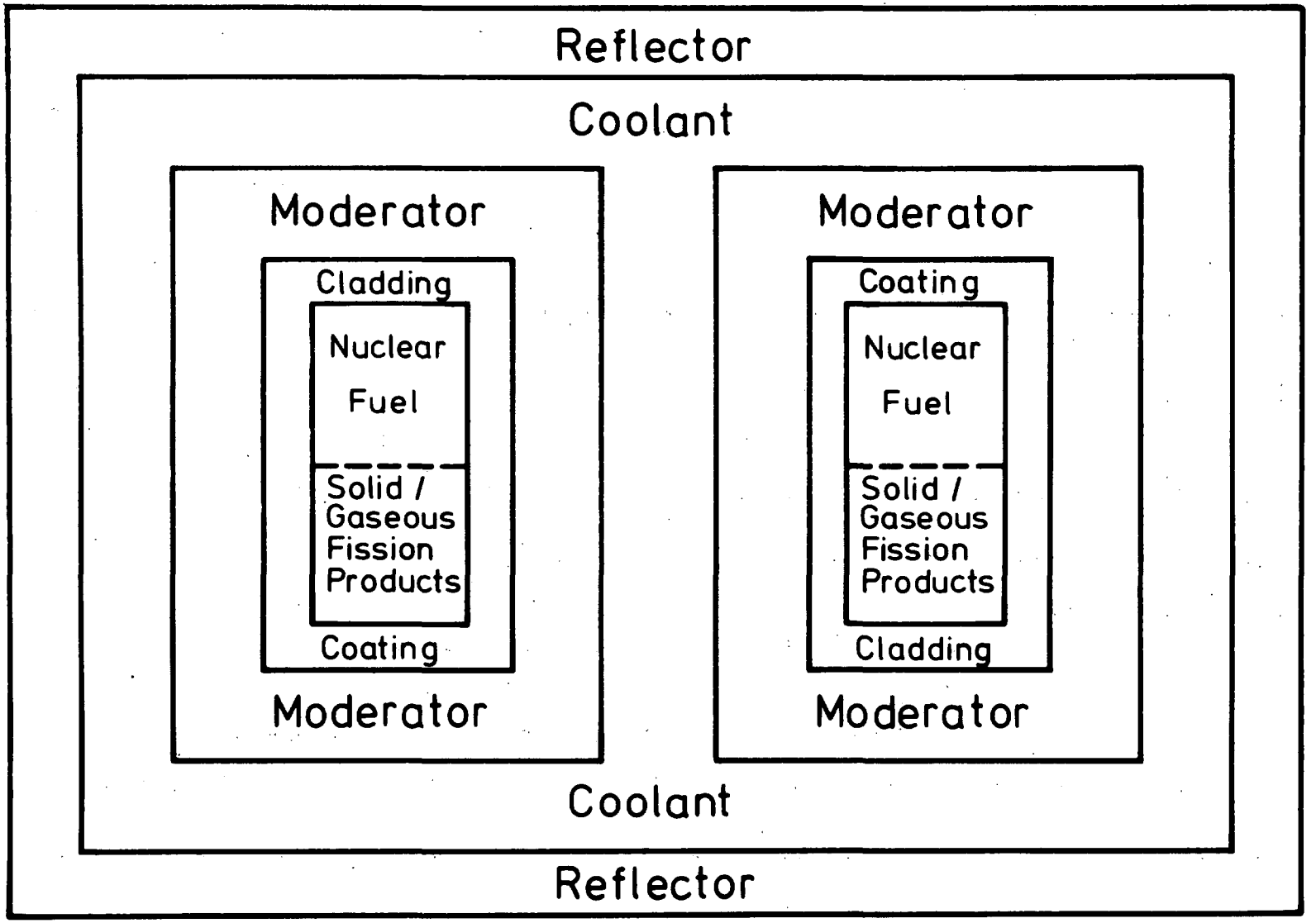

Fig. 1:
Scheme of thermal reactor fuel element components

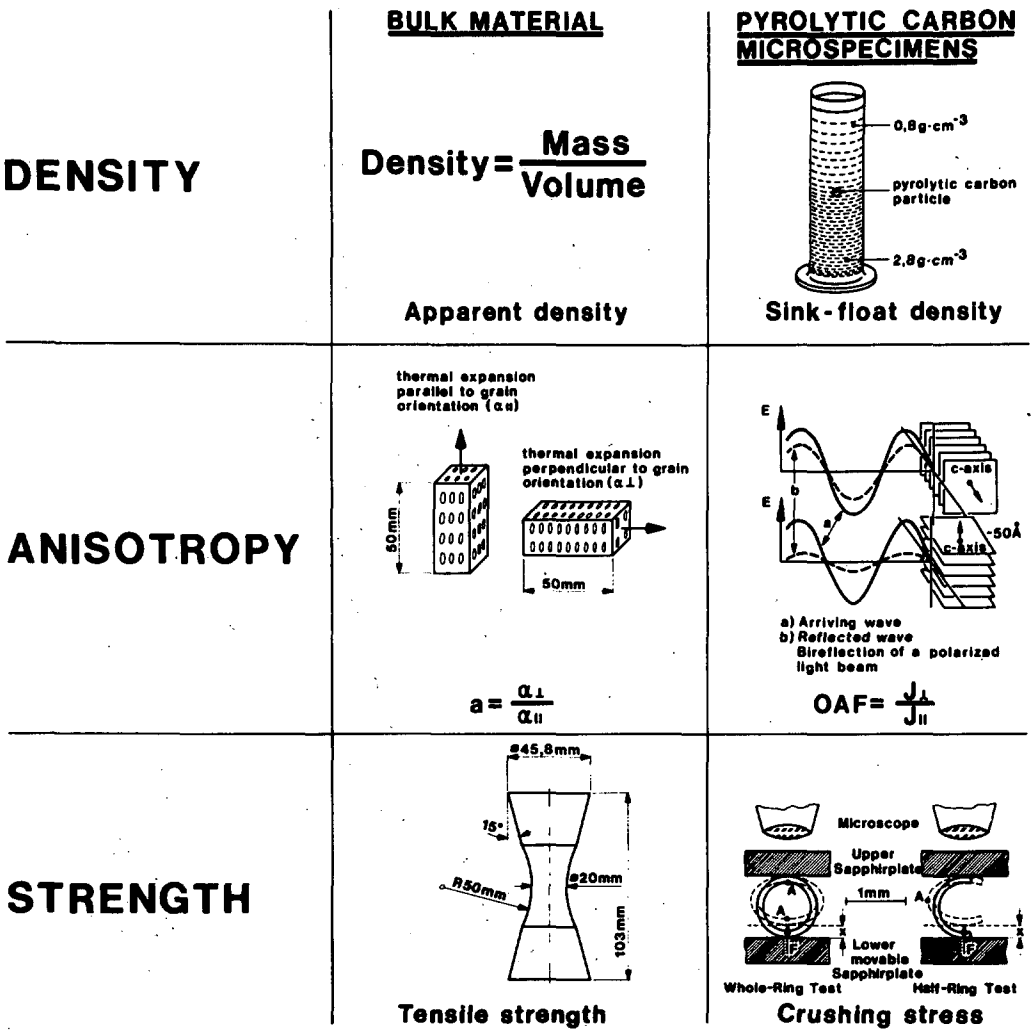

Fig. 2:
Comparison of methods for the determinations of density, anisotropy and strength measured on bulk materials and pyrolytic carbon microspecimens

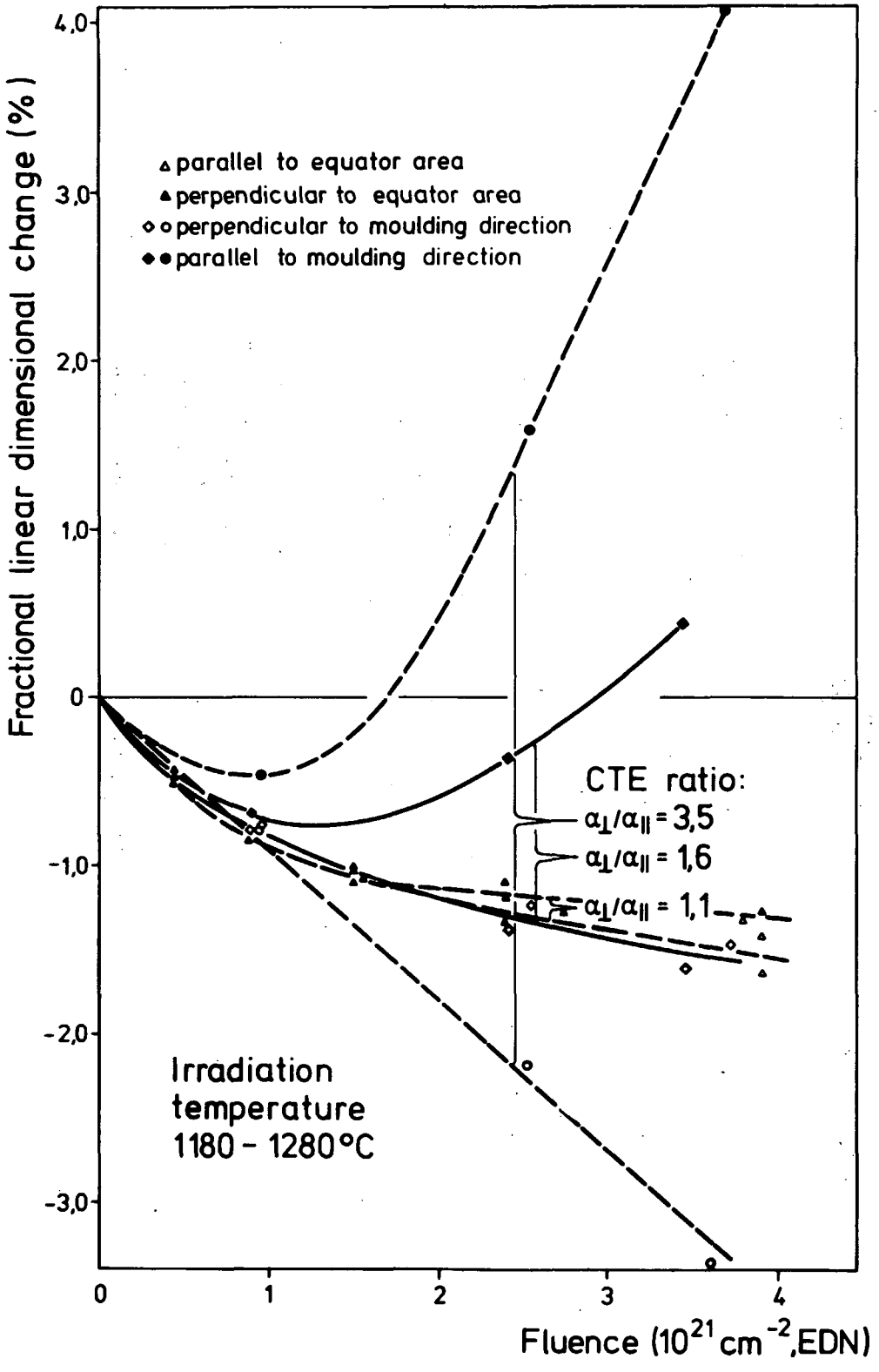

Fig. 3:
Influence of anisotropy of graphitic matrix materials on the irradiation induced dimensional change (25)

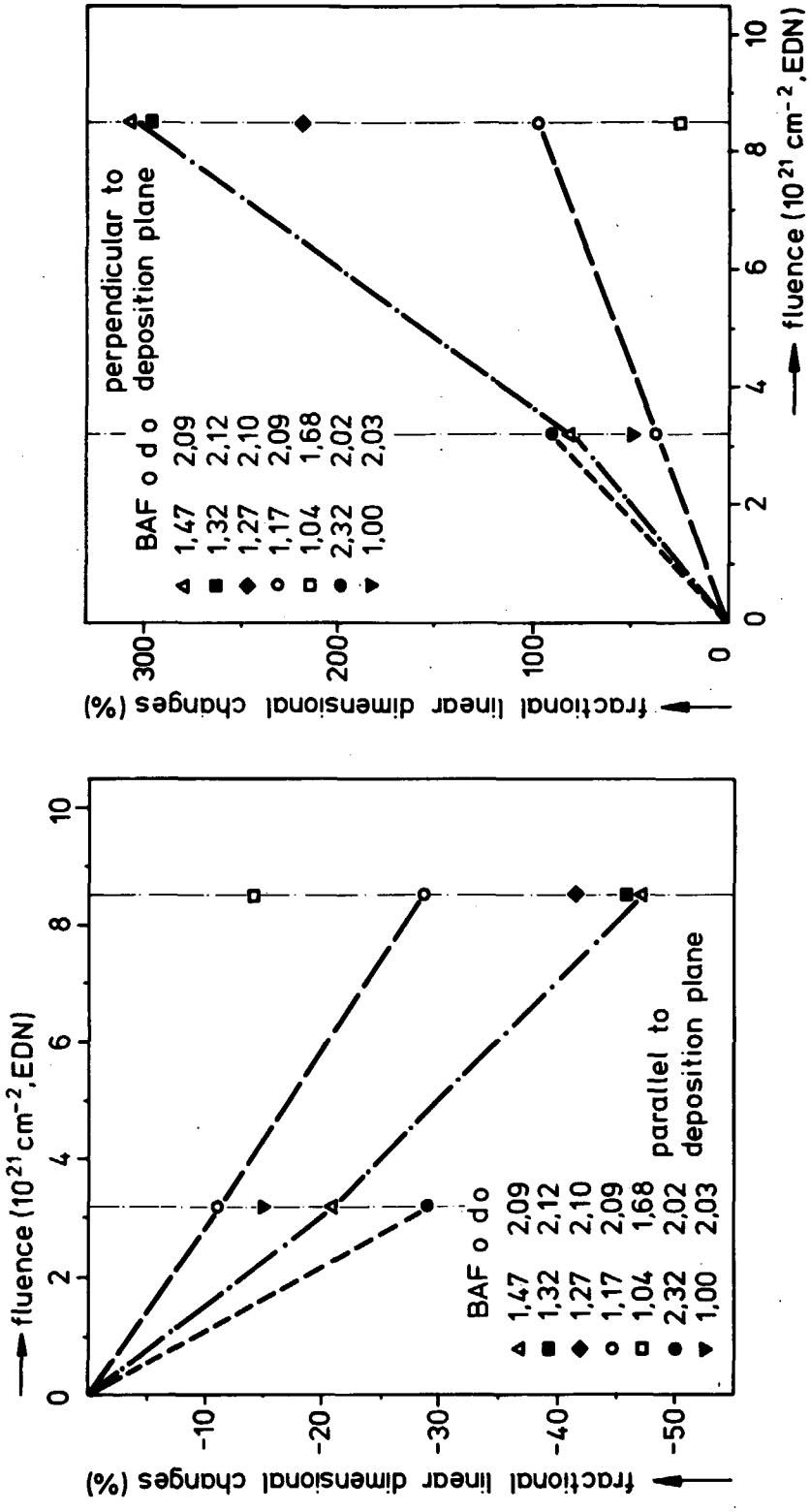

Fig. 4:
Influence of orientation anisotropy on the irradiation induced dimensional changes of unstressed pyrolytic carbon deposited from methane on graphite disks (not on fuel kernels) (27)